MISSOURI DEPARTMENT OF NATURAL RESOURCES
DIVISION OF ENVIRONMENTAL QUALITY
ENVIRONMENTAL SERVICES PROGRAM
Standard Operating Procedures

SOP #: MDNR-FSS-106 EFFECTIVE DATE: February 10, 2004

SOP TITLE: Field Analysis of Flash Point

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SUMMARY OF REVISIONS: Format changes were made to conform to new SOP procedures.

APPLICABILITY: Applies to the analysis of flash point conducted in the field by the EER/FS Section of the Environmental Services Program

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RECERTIFICATION RECORD:

Date Reviewed				
Initials				

1.0 SCOPE AND APPLICABILITY

The procedures contained in this document are applicable to ESP field personnel who are tasked with the analysis of samples for flash point using the Setaflash Closed Cup Tester (hereafter Setaflash).

2.0 SUMMARY OF METHODS

The analysis of flash point described below is a relatively simple and easy method for determining the ignitability characteristics of a hazardous waste, as defined in 40 CFR Part 261.21, or a hazardous material such as a flammable liquid, as defined in 49 CFR Part 173.120. The following method is specific to the Setaflash. The test involves heating a small amount of liquid material and applying a flame to the head space. The process is continued until the temperature is reached at which the head space ignites. The flash point test can be somewhat subjective, as the flame of some materials is very difficult to see.

3.0 DEFINITIONS AND ACRONYMS

- ASTM - American Society for Testing and Materials
- Flash point - The temperature at which a liquid gives off vapor sufficient to form an ignitable mixture in the air near the surface of the liquid.
- HASP - Health and Safety Plan
- HAZWOPER - Hazardous Waste Operations and Emergency Response
- HSERP - Hazardous Substance Emergency Response Plan
- PPE - Personal Protective Equipment
- Safety Officer - The person, generally the ESP sampler, who is assigned or assumes the duties of the health and safety officer for a specific investigation.
- Setaflash Closed Cup Tester - A laboratory/field instrument used for conducting closed cup flash point tests.
- Target temperature - The regulatory flash point temperature or predetermined temperature of concern.

4.0 HEALTH AND SAFETY WARNINGS

Due to the nature of materials that may be tested and the fact that these materials will be heated as part of the analytical procedure, caution should be taken with respect to fumes and explosion. The following precautions should always be taken:

4.1 The level of personal protection required for conducting field flash point tests shall be described in the site specific HASP and must be discussed in the site safety briefing by the Safety Officer. In the absence of a HASP, gloves and safety glasses should always be worn as minimum PPE.

4.2 The Setaflash should always be used under a fume hood or in a well-ventilated area. The fume hood must be turned off while the actual test is being conducted, however, so as not to blow out the flame. The hood should be turned on between samples to exhaust any fumes.

4.3 A fire extinguisher should always be within easy reach when using the Setaflash.

5.0 PERSONAL QUALIFICATIONS

All ESP personnel directly involved in field investigations at sites that fall under the EPA Worker Protection requirements of 40 CFR Part 311, referencing OSHA 29 CFR Part 1910.120, and meet the definition of HAZWOPER activities must:

- attend a 40-hour course designed to meet the OSHA health and safety training requirements for hazardous site workers;
- attend an annual 8-hour health and safety refresher course, or receive equivalent training;
- participate in the departments medical monitoring program;
- receive appropriate on-the-job training;
- be familiar with the HSERP, written and maintained by the ESP; and
- be familiar with all ESP SOP documents that are applicable to the field activities, including but not limited to those referenced in this SOP.

6.0 SUPPLIES AND EQUIPMENT

- A Setaflash Closed Cup Tester as described in ASTM Method D3278-78 or D3828-93.

- A glass syringe of 2 ml capacity and a thermometer with a scale error that does not exceed 0.25^{o}C (0.5^{o}F) will be supplied with the instrument
- Absorbent paper tissue for cleaning cup between uses
- Match or lighter

7.0 REAGENTS

- P-Xylene for calibration reference, low-end standard 26.4-28^{o}C, and glacial acetic acid calibration reference, high-end standard 39-41^{o}C (calibration references may be obtained from the Chemical Analysis Section)
- Cooling mixture of ice water or dry ice
- Liquefied petroleum gas
- Appropriate solvent, detergent, or other cleaning agent for cleaning the cup *(NOTE: Solvents may include methanol, methylene chloride, hexane, or acetone, however many of these are flammable and could potentially skew the test results. A detergent such as Alconox, or other non-flammable cleaning compounds such as Simple Green may be more appropriate.)*

8.0 HANDLING AND PRESERVATION

8.1 Obtain at least a 25 ml sample from the bulk source and store in a nearly full, tightly closed glass container suitable for the type of liquid being sampled (e.g. VOA vial or 2 oz jar).

8.2 Do not use plastic containers since some volatile compounds can diffuse through the walls.

8.3 The sample should be kept cool on ice prior to analysis.

8.4 Avoid loss of volatile materials from the sample, which may cause erroneously high flash points. Do not open sample containers unnecessarily or transfer sample to the Setaflash cup unless its temperature is at least 10^{o}C (20^{o}F) below the expected flash point. Discard samples from leaking containers.

9.0 PROCEDURE

9.1 The manufacturer's operating and maintenance instructions should be read for proper care and servicing of the Setaflash.

9.2 Prior to beginning the actual test, determine the relationship between the Setaflash's temperature control dial and thermometer readings at intervals of 5^{o}C. This will help the analyst know the approximate amount of dial adjustment that will be necessary to make a 5 ^{o}C increment change.

9.3 The Setaflash should be placed in subdued light and out of interfering drafts. Provide a dark shield if necessary and turn off the exhaust fan if used.

9.4 Insure that the inside of the cup, lid, and shutter mechanism are clean and free of contamination. If necessary, wipe clean with an absorbent tissue, using

solvent or detergent if needed, and lock the lid tightly in place. If flammable solvents are used for cleaning, insure that they have completely evaporated prior to conducting the analysis.

9.5 To check the accuracy of the Setaflash, determine the flash point of either p-xylene or glacial acetic acid reference standard, depending on the temperature range of interest. In some instances, for example when finite flash point temperatures are being determined, the analyst may wish to use both standards. An average of 27.2 ^{o}C ± 0.8 for p-zylene and 40 ^{o}C ± 1.0 for glacial acetic acid should be obtained. If not, check that sufficient heat transfer paste surrounds the thermometer to provide good heat transfer from the cup.

9.6 Flash/no flash operation from: Ambient to 110^{o}C (230^{o}F)

9.6.1 Switch the Setaflash on by placing the switch to either the "power" or "battery" position, depending on the power source (110V AC or 12V DC). To increase the temperature to the target temperature, turn the heat control dial clockwise. A red light on the face of the instrument will glow when the cup is heating. When a temperature of about 3^{o}C below the target temperature is reached, slowly turn the dial counterclockwise until the light just goes out. The temperature of the cup is stable when the light slowly cycles on and off.

9.6.2 Determine the barometric pressure to obtain the corrected target temperature at that pressure (see section 9.11). If a barometer is not available, the barometric pressure can be obtained from any local weather service or radio station. Most weather services provide the barometric pressure in inches of mercury rather than millimeters. To convert inches to millimeters, multiply by 25.4.

9.6.3 When the temperature has stabilized at the target temperature, charge the 2 ml syringe with the sample to be tested and transfer it to the filling orifice located on the cup lid. Discharge the sample into the cup by completely depressing the syringe plunger. (NOTE: Disposable pipettes may be used in place of the 2 ml syringe to avoid having to decontaminate the syringe between samples.)

9.6.4 Immediately after injecting the sample into the cup, set the time device by rotating it to the one-minute mark. While the timer is counting, open the gas control valve, light the pilot and adjust the test flame with the pinch valve to match the size of the flame gauge, the 0.157 inch (4 mm) gauge ring located on the top of the cup lid.

9.6.5 When the timer bell rings, indicating that one minute has elapsed, quickly observe the temperature and apply the test flame. The test flame is properly applied by opening the slide fully and closing completely over a period of approximately 2 seconds while watching for a flash.

(NOTE: If a comparatively large blue flame that propagates itself over the surface of the liquid is observed, the sample is considered to have flashed.)

9.6.6 Turn off the pilot flame and clean the apparatus in preparation for the next test (see section 9.10).

9.7 Flash/no flash operation from: $0^{o}C$ ($32^{o}F$) to ambient

9.7.1 If the target temperature is at or below the ambient temperature, the cup and the sample should be cooled to 5 to $10^{o}C$ below that point by using ice or dry ice.

9.7.2 Introduce the sample as in 9.6.3. Allow the temperature to rise under ambient condition or increase the temperature of the cup by use of the heater control. Determine whether the sample flashes as in 9.6.4 and 9.6.5. (NOTE: Occasionally, if near the actual flash point temperature, application of the test flame may give rise to a halo; this should not be interpreted as a flash.)

9.7.3 Turn off the pilot flame and clean the apparatus (see section 9.10).

9.8 Finite flash point: Ambient to $110^{o}C$ ($230^{o}F$)

9.8.1 A preliminary or trial test should be run at an estimated finite flash point. Follow steps 9.6.1 to 9.6.5.

9.8.2 If a flash is observed, proceed as follows:

- Repeat 9.6.5 using a temperature of $5^{o}C$ below the original estimated finite flash point. If a flash is still observed, repeat at $5^{o}C$ lower intervals until no flash is observed. (NOTE: Never make repeat tests on the same sample aliquot. Use a fresh 2 ml of the sample for each repeat test.)
- Starting at the temperature at which no flash occurred, increase the temperature at $0.5^{o}C$ intervals and repeat step 9.6.5. At least one additional duplicate test should be run when determining finite flash point. Record the temperature at which the flash actually occurs (see 9.12.2)

9.8.3 If no flash is observed in 9.8.1, proceed as follows:

- Using a temperature of $5^{o}C$ higher than that observed in 9.8.1, repeat step 9.6.5. If no flash is observed, repeat at $5^{o}C$ intervals until a flash is observed.
- Starting at the last $5^{o}C$ interval temperature at which no flash occurred, increase the temperature at $0.5^{o}C$ intervals and repeat step

9.6.5. At least one additional duplicate test should be run when determining finite flash point. Record the temperature at which the flash actually occurs (see 9.12.2).

9.9 Finite flash point: 0°C (32°F) to ambient temperature

9.9.1 A preliminary or trial test should be run by cooling the sample to 3 to 5°C below the estimated finite flash point.

9.9.2 Using ice water or dry ice, cool the Setaflash cup to approximately the same temperature as the sample.

9.9.3 Determine the finite flash point as in steps 9.8.2 and 9.8.3.

9.10 Cleanup of Setaflash and preparation for next test

9.10.1 To clean the cup, unlock the lid assembly of the Setaflash cup and raise to the hinge stop. Soak up the sample with an absorbent paper tissue and wipe the cup dry. Clean the under side of the lid and filling orifice. A pipe cleaner may be helpful in cleaning the orifice.

9.10.2 A suitable solvent or detergent (see 7.0 REAGENTS) may be needed if the liquid tested was viscous, such as oil or tar materials, or contained dispersed solids that may not be easily cleaned from the cup.

9.10.3 After cleaning the cup and to save analytical time, the temperature may be increased to some standby value by adjusting the heat control dial.

9.10.4 The syringe may be easily cleaned by filling it several times with the cleaning solution, discharging the solution every time and allowing the syringe to air dry with the plunger removed. See MDNR-FSS-206 *Decontamination Procedures for Sampling Equipment*.

9.11 Barometric pressure correction:

9.11.1 When the barometric pressure differs from 760 mm Hg, calculate the flash point temperature using the following equations:

$$\text{Calculated flash point} = C + 0.03\,(760 - P)$$
$$= F + 0.06\,(760 - P)$$

where;

C, F = observed flash point temperature (°C or °F)

P = barometric pressure (mm Hg)

9.11.2 The corrected target temperature can also be determined by the following equation:

$$C = S - 0.03 (760 - P)$$
$$F = S - 0.06 (760 - P)$$

where;

C, F = flash point temperature to be observed to obtain the target temperature at standard pressure

S = target temperature at standard pressure

9.12 Report:

9.12.1 When using the flash/no flash method, report whether the sample flashed at the required flash point and the method used (e.g. flash/no flash: ambient to 110^{o}C).

9.12.2 If a finite flash point was determined, report the average of duplicate runs to the nearest 0.5^{o}C.

10.0 QUALITY ASSURANCE AND QUALITY CONTROL

10.1 All of the quality control data should be noted in the field log book (see MDNR-FSS-004 *Field Documentation)* and available for review.

10.2 Duplicates and standard reference materials should be routinely analyzed.

10.3 The flash point of the p-xylene and/or glacial acetic acid reference standard must be determined on a duplicate at least once per sample batch. The average of the two analyses should equal 27.2 ^{o}C $\pm$ 0.8 for p-zylene or 40 ^{o}C $\pm$ 1.0 for glacial acetic acid.

11.0 REFERENCES

- ASTM Method D3278-78 or D3828-93
- MDNR Field Services Section standard operating procedures
- 40 CFR Part 311
- 40 CFR Part 261